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STRUCTURE, PHASE TRANSFORMATIONS, AND DIFFUSION

Structure of Diffusion Layers Formed at Liquid Aluminum Alloy–Steel Contact Boundary

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Abstract—The microstructure and composition of diffusion layers that arise upon the spread of the liquid aluminum alloys AL5, AL9, AL852, and A7075 over the St3 steel has been investigated using optical and electron microscopy. The thermophysical conditions of the interaction of the melt with the substrate under which at the boundary of the substrate the plastic transition layer of iron-based solid solution improving quality of the coating is formed have been determined.

Keywords: melt, diffusion layers, steel, structure, phase composition

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INTRODUCTION

The diffusion transition layers between the aluminum melt and steel are formed due to the chemical interactions at the Al melt–steel contact boundary [1–4]. The intense dissolution of steel in the melt is accompanied by the formation of crystals of intermetallic Al_xFe_y compounds. Since the presence of these phases substantially reduces the strength of compounds and worsens the quality of coatings [1], the search for ways to improve the technology of applying corrosion-resistant coatings onto steel and enhancing their properties remains topical.

This work is a continuation of investigations by the authors on studying the structure of diffusion layers that arise upon the spread of binary aluminum melts over steels. In the previous paper [5], the experimental procedure and features of the formation of a three-layer composite upon the spread of Al–Si melts over the steel substrate were described in detail; it was shown that the temperature–time regime of the formation of diffusion layers markedly affects their phase composition and structure. In particular, an increase in the time of contact between liquid and solid metals from 15 to 120 min at 1200°C resulted in the formation of a more chemically homogeneous coating due to the fact that the intermediate plastic interlayer that represents an iron-based solid solution arises between the intermetallic layer and substrate. It was of interest to check the possibility of the appearance of this plastic interlayer when liquid binary silumins are replaced by multicomponent Al alloys.

The purpose of this work was to study the structure and phase composition of diffusion layers formed at the contact surface between multiphase aluminum alloys and the St3 steel.

EXPERIMENTAL

Compositions of alloys used as coatings on the steel substrate are listed in the table. The first three compositions are related to cast Al–Si–Cu–Mg alloys, and the last composition corresponds to a high-strength wrought alloy of the Al–Zn–Mg–Cu system.

Table

Grade of alloy	Weight fraction of elements, %								
	Si	Fe	Cu	Mg	Zr	Ni	Mn	Zn	Ti + Zr
AL5	4.5–5.5	to 0.4	1.0–1.5	0.35–0.55			to 0.1		to 0.15
AL9	6.0–8.0	to 0.5	to 0.2	0.2–0.4			to 0.5		to 0.15
AL852	6.25		2.0	0.75		1.2			
A 7075	0.4	0.5	1.2–2.0	2.1–2.9			0.3	5.1–5.6	

Multilayer samples were obtained in vacuum at 1×10^{-3} Pa under the conditions of separate preliminary heating of the aluminum melt and steel. An Al alloy weight was placed into the graphite container of a dosing unit, next heated to a temperature of 700°C, and finally extruded onto the steel substrate. Then, the bimetallic sample was slowly ($\sim 10^{-6}$ K/s) heated to 1200°C. Upon crystallization, the cooling rate was ~10 K/s. Regimes with these rates provided the long-term (not less than 2 h) contact of aluminum melts with the substrate. The metallographic study of macro- and microstructures of the crystallized samples and their phase analysis were performed with MBS-9 and Neophot-32 optical microscopes and a Quanta-200 scanning electron microscope (SEM) with an attachment for electron microprobe analysis (EMA). To measure microhardness, we used a PMT-3 device at a load of 2 N (measuring error 5%), and the average values of microhardness were calculated on the basis of ten measurements.

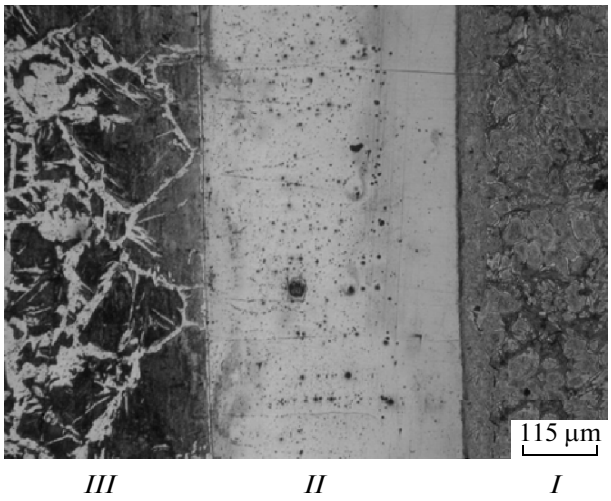

Fig. 1. Microstructure of diffusion layers formed upon spread of A7075 melt over St3 steel: (*I*) surface layer, (*II*) transition layer, and (*III*) substrate; SEM.

RESULTS AND DISCUSSION

When the contact interaction of the melt with the steel was investigated visually, it was found that the characteristic crater was formed under the molten drop, which was caused by the dissolution of the substrate material in the melt. The metallographic analysis of the cross section of the aluminum alloy–steel contact boundary showed that, due to the spread of Al melts over the steel substrate, a layered composite material was formed. Typical microstructure of the cross section of the layered sample (using alloy AL5 as an example) is depicted in Fig. 1. The microstructure is characterized by three zones, i.e., (*I*) a surface layer, (*II*) a transition layer, and (*III*) the substrate. To determine the phase composition of layers formed under the given experimental conditions, the microhardness of the composite was measured and, using the EMA method, the distribution of concentrations of all the elements (Fe, Al, Mg, Mn, Cr, Cu, and Si) that are included into the alloy composition was determined over the cross section of layered samples.

As follows from Fig. 2, plots of changes in the hardness over the cross section of diffusion layers for all compositions differ very little. This indicates the general laws of the dissolution of iron in Al melts and the formation of layers of close compositions. To study these laws, the evolution of the composite structure within each diffusion layer was considered.

Structure and Composition of Surface Layers

The thickness of surface layers in all samples is 1500–2500 μm. Over the entire surface of outer layers macrodefects are observed in the form of cracks and pores. The presence of these defects causes the nonmonotonic distribution of H_v over the cross section of the samples (Fig. 2). The measured values of microhardness over the cross section of the layer proved to be within $6000 < H_v < 7000$ MPa, which, according to values of hardness in Al–Fe intermetallic compounds known in the literature, corresponds to the AlFe composition [6, 7]. The most homogeneous and defect-free layer with equal hardness 6500 MPa over the cross section is formed in the steel St 3 + alloy AL5 sample.

The most typical structure of the crystallized surface layer shown in Fig. 3 is illustrated by the steel St 3 + alloy A7075 composite. The layer consists of grains with dendrite morphology (Fig. 3a) between which lower-melting plate-type eutectics is located (Fig. 3b). A comparison of the elemental compositions of structural constituents established by the EMA data has shown that, in the region of grains, the amount of iron and aluminum is nearly the same, i.e., 45–50 at %; in other words, the above results of hardness measurements were confirmed. The interdendritic space is enriched with aluminum (to 60 at %) and other low-melting elements (zinc, magnesium). At this aluminum content, according to [6, 7], the eutectics composition can contain intermetallic compounds with variable compositions from FeAl to $FeAl_2$. The average width of eutectic plates is 0.1–0.2 μm (Fig. 3b), and their composition hardly differs from that of grains. Due to this disperse structure, microhardness of this structural constituent is $H_v =$ 8000 MPa, which is higher than the average microhardness of the layer by 1500 MPa.

As was established from the EMA results, the main distinction of the phase composition of surface layers that arise upon the crystallization of silicon-bearing melts (AL852, AL5, AL9) is the fact that silicon is included into the iron aluminide composition to form the Al_8Fe_8Si-type ternary phase. Figure 4a displays a section of the surface layer of the steel St 3–alloy AL852 sample. The surface-layer microstructure is

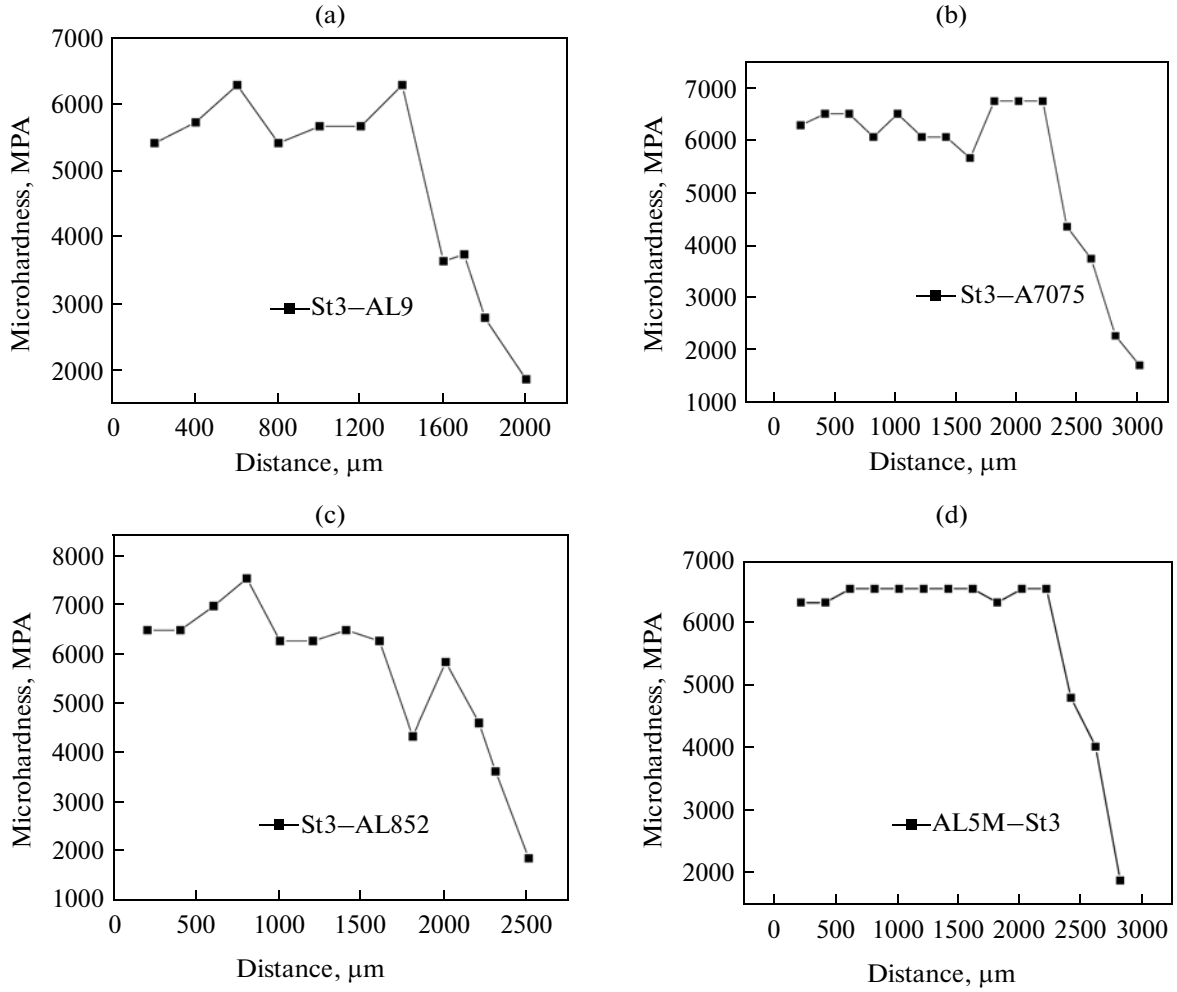

Fig. 2. Microhardness distribution over cross section of surface and transition layers in composites of different chemical composition: (a) AL9, (b) A7075, (c) AL852, and (d) AL5.

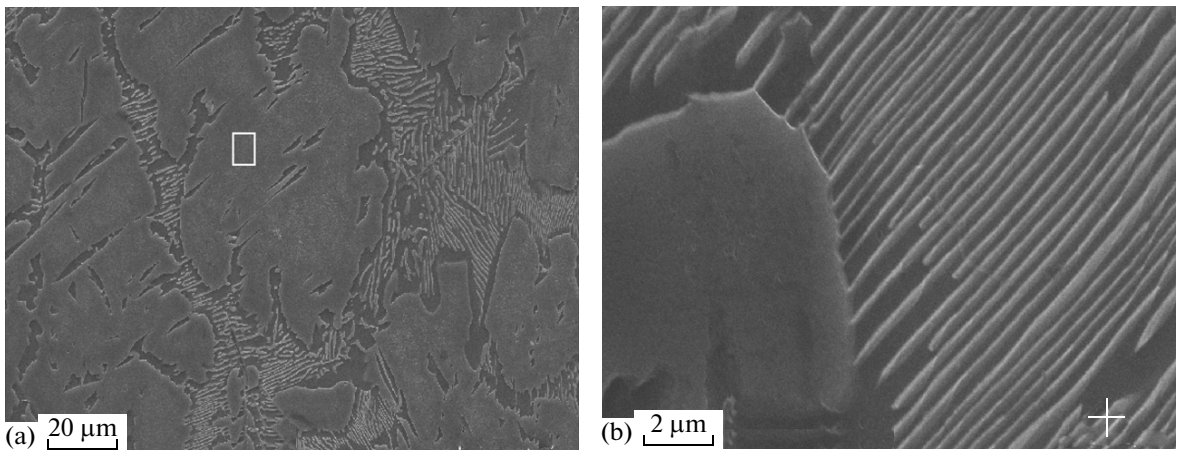

Fig. 3. Microstructure of surface layer in steel St 3–alloy A7075 sample (SEM): (a) grains of dendrite morphology and (b) plate-type eutectics.

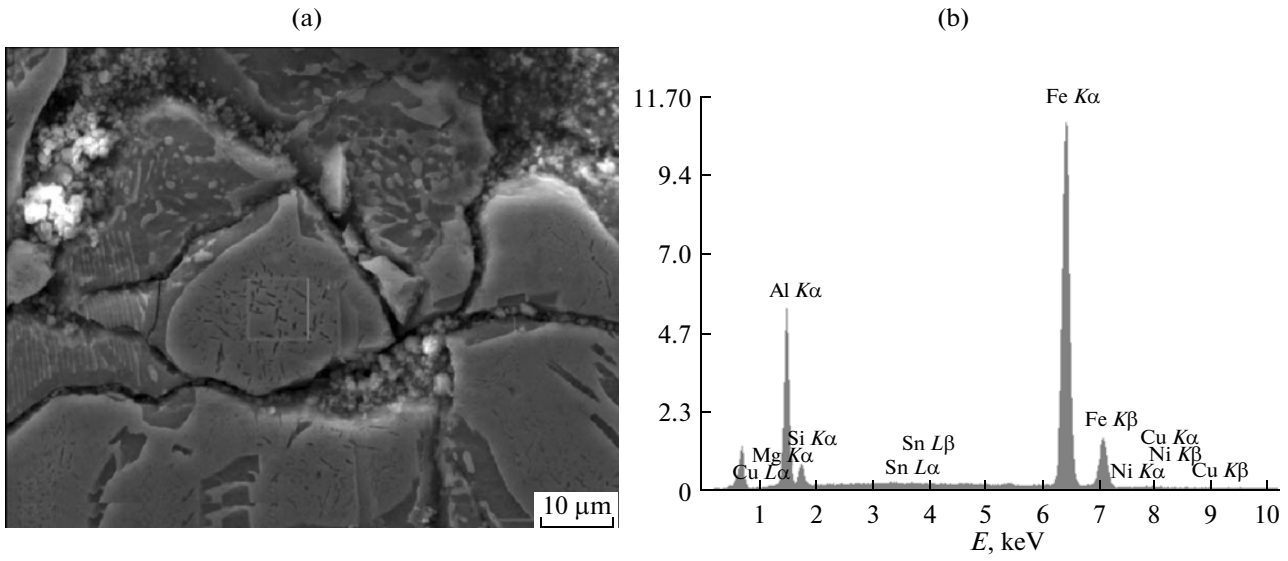

Fig. 4. (a) Microstructure and (b) chemical composition of the surface layer in steel St 3–alloy AL852 sample (SEM).

also inhomogeneous, and the phase with grains 30–50 μm in size occupies the main volume. The multiphase eutectics of segregation origin with cellular and plate-type structures are located along the grain boundaries depending on the cross section of colonies. In addition to iron and aluminum, alloying elements of initial alloys were found in the aluminide composition, i.e., copper and magnesium were present in amounts no larger than 0.5–1.0 at % (Fig. 4b).

Structure and Composition of Transition Layers

In all samples, the transition layer is easy to discern visually (Fig. 1) and, depending on the composition of the melt in contact with the steel, its width changes from 500 μm in the steel St 3–alloy AL9 and steel St 3–alloy AL852 samples to 300 μm in the steel St 3–alloy AL5 sample. Measurements of the concentration of elements included into alloy compositions using the EMA method have shown that, in all transition layers, the amount of iron decreases monotonically and the amount of aluminum increases in the direction from the substrate to the surface layer. For example, in the steel St 3–alloy AL9 composite, the average concentrations of iron and aluminum are 77 and 22 at %, respectively. Previously, the same experimental data were obtained by the authors when studying features of the formation of diffusion layers that arise upon spread of binary aluminum–silicon melts [5]. Based on these results, it can be assumed that the transition layer is formed near the substrate due to the reaction diffusion of iron into aluminum and represents an iron solid solution with variable aluminum concentration. The other alloying elements (Mg, Mn, Cr, Cu, and Si) are distributed rather uniformly across the layer thickness. Changes in the chemical composition of the composite across the thickness of the transition layer manifest themselves graphically in dependences of its microhardness. In particular, an enhancement in the iron concentration from the boundary with the surface layer to the boundary with the substrate results in gradual approach of hardness of the layer to that of the steel (Fig. 2).

Structure and Composition of the Substrate

Near the transition layer, the substrate exhibits the structure of annealed steel St3 and consists of ferrite with microhardness $H_v \sim 1750–1800$ MPa and pearlite (Fig. 5). The narrow (2500 μm) band adjacent to

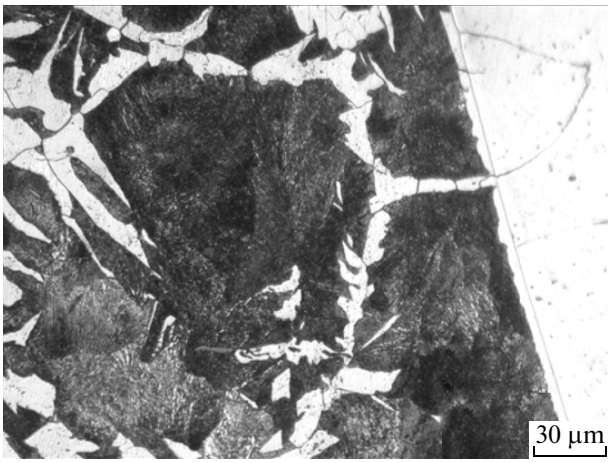

Fig. 5. Microstructure of substrate in steel St 3–alloy AL9 sample.

the transition layer has a ferritic–pearlitic structure (coarser as compared to the main volume), and the cause of its formation is the heating of the substrate in the process of experiments.

CONCLUSIONS

Based on the experimentally determined compositions and hardness of diffusion layers that arise upon the spread of superheated multicomponent aluminum melts over the steel-substrate surface, it was established that, due to the chemical interactions at the Al melt–steel contact surface, the intense dissolution of the substrate occurs. Upon the supersaturation of the liquid aluminum alloy with iron and the subsequent crystallization, intermetallic compounds of complex chemical composition based on equiatomic AlFe aluminide take place. These results are in full agreement with the available literature data [1–5]. The width of these layers is on the order of 2000 μm, and the hardness is 6500–7000 MPa. As was noted in the introduction, the presence of this brittle layer between the coating and substrate unfavorably affects the quality of articles and their service properties. The investigations performed in this work have shown that, in experiments, when the temperature–time regime changes due to an increase in the time of contact between solid and liquid phases to 120 min at 1200°C, between the steel substrate and aluminum coating the additional plastic diffusion layer is formed, whose intermediate composition and lower values of hardness make it possible to predict the increased strength of joint of two metals and the improved quality of the coatings under study.

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